LONG WET STAND SILVER OXIDE-ZINC CELL TESTS (PHASE I)

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LONG WET STAND SILVER OXIDE-ZINC CELL TESTS (Phase I)

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AIR FORCE AERO PROPULSION LABORATORY
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This report contains the results of an effort to increase the activated shelf life of silver oxide-zinc cells through the use of improved separator materials. The work was performed in the Aerospace Power Division of the Air Force Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson AFB, Ohio under Project 3145, Task 22, and Work Unit 39. The effort was conducted by Michael P. Dougherty, AFAPL/POE-2 during the period July 1971 to June 1975.

This report has been reviewed by the Information Office (ASD/OIP) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

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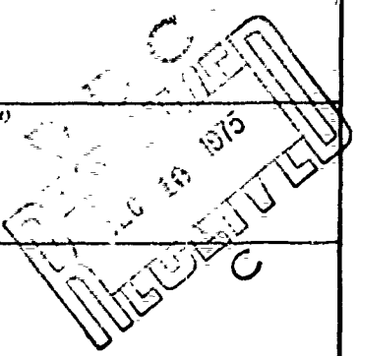

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Specially built twenty ampere-hour, silver oxide-zinc cells were stored wet and charged at different temperatures for up to two years to determine capacity retention as a function of storage time and separator material. A total of 505 cells were fabricated using two to four layers of RAI P-2291 (40/60) or four layers of Fibrous Sausage Casing (FSC) as the separator material. Prior to storage, the cells were electrically conditioned and their capacities measured. Cells with each type of separator were then stored at 70°F, 115°F, and 150°F and their capacities checked periodically.

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Cells fabricated with three and four layers of RAI P-2291 as the separator material performed as well as those fabricated with four layers of FSC with respect to capacity retention with storage. Because of the difference in material thickness, however, the cells with RAI separators yield a 23% to 26.5% increase in volumetric energy density (watt-hr/cu.in) over the standard FSC.

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SECTION I

CELL DESCRIPTION

A total of 505 specially fabricated silver oxide-zinc cells were purchased during the period July 1971 through October 1971 from Eagle-Pitcher Industries, Inc., (Contract F33615-71-C-1243). The cells were a standard commercial package size with a nominal capacity of 20 A-H, and contained a vent valve with opening pressure in the range of 2 to 20 psig. The positive plates were pure silver on an expanded silver grid. The negative plates were terminated zinc oxide with 3% by weight mercuric oxide also on an expanded silver gird. The negative plates in all cells were unwrapped, while all positive plates were wrapped with one layer of Webril (E 1408). The electrolyte was 45% by weight potassium hydroxide.

In addition to the one layer of Webril, 135 cells had positive plates wrapped with two (2) layers of RAI P-2291 (40/60), 135 with three (3) layers of RAI P-2291 (40/60), 135 with four (4) layers of RAI P-2291 (40/60), and 100 with four (4) layers of fibrous sausage casing (FSC). Cells with less than the control thickness of separation (4 layers of FSC) were shimmed to maintain the same cell tightness factor. A detailed cell description is given in Appendix I.

Table 1 gives the serial numbers and electrolyte volumes corresponding to each separator configuration. The difference in electrolyte volume is due to the difference in separator thickness and the resulting void volume.

TABLE 1

RELATIONSHIP OF S/N TO SEPARATOR TYPE AND ELECTROLYTE VOLUME

S/N	Separation	Electrolyte Vol. (45% KOH)
1 - 100	4 Layers FSC	65cc
101 - 235	2 Layers RAI	45cc
236 - 370	3 Layers RAI	45cc
371 - 505	4 Layers RAI	45cc

SECTION II

TEST PROCEDURE

The cells were distributed into twenty-five (25) groups for long wet stand testing. A detailed breakdown relating group numbers to serial numbers and separator configuration is shown in Table 2. Twenty (20) groups contained five (5) cells of each separator configuration (20 cells/group) while five (5) groups contained all but the FSC separation (15 cells/group). No cells with FSC separators were included in groups 1, 2, 3, 8 and 9 because of data already existing in the literature indicating that AgO-Zn cells with this separator configuration would not stand under the conditions to be experienced by these cells.

In addition, cell S/N's 77, 78, 191, 318 and 462 were received in damaged condition. Cells 318 and 462 were replaced with cells 361 and 496, respectively. Cell 191 was not replaced, which left group 5 containing only four (4) cells with two (2) layers of RAI separation. Cells 77 and 78 could not be replaced because no extra cells with FSC separation were available. Therefore, group 6 contained only three (3) cells with FSC separation.

Working with one group at a time, the cells were then vacuum filled with electrolyte and allowed to stand overnight at room temperature. Following this stand, the cells were then subjected to several conditioning cycles consisting of a two ampere constant current charge for sixteen (16) hours and a 20 ampere constant current discharge. The cells were

limited to 2.05 volts on charge and were discharged to 1.20 volts. Cycling continued until the capacity stabilized and was the same for two consecutive cycles. On the average, five conditioning cycles were required for each group.

Once conditioned, the cells were recharged and placed in storage at the temperature and for the time periods specified in Table 3. However, after approximately 45 days of storage, it was noted that the epoxy on cells 88 and 89 (group 12) and cell 427 (group 16) was softening, and there was a large buildup of electrolyte around the relief valve. The storage temperature for groups 12, 13 and 16 was, therefore, reduced to 150°F, and all subsequent high temperature storage was done at 150°F.

TABLE 2

RELATIONSHIP OF S/N TO GROUP NUMBER AND SEPARATOR TYPE

Group	4 Layers FSC	2 Layers RAI	3 Layers RAI	4 Layers RAI
1	None	221 - 225	356 - 360	491 - 495
2	None	216 - 220	351 - 355	486 - 490
3	None	211 - 215	346 - 350	481 - 485
4	96 - 100	201 - 205	336 - 340	471 - 475
5	91 - 95	191 - 195	326 - 330	461 - 465
6	76 - 80	176 - 180	311 - 315	446 - 450
7	61 - 65	161 - 165	296 - 300	431 - 435
8	None	206 - 210	341 - 345	476 - 480
9	None	196 - 200	331 - 335	466 - 470
10	81 - 85	181 - 185	316 - 320	451 - 455
11	66 - 70	66 - 170	301 - 305	436 - 440
12	86 - 90	186 - 190	321 - 325	456 - 460
13	71 - 75	171 - 175	306 - 310	441 - 445
14	46 - 50	146 - 150	281 - 285	416 - 420
15	51 - 55	151 - 155	286 - 290	421 - 425
16	56 - 60	156 - 160	291 - 295	426 - 430
17	31 - 35	131 - 135	266 - 270	401 - 405
18	36 - 40	136 - 140	271 - 275	406 - 410
19	41 - 45	141 - 145	276 - 280	411 - 415
20	16 - 20	116 - 120	251 - 255	386 - 390
21	21 - 25	121 - 125	256 - 260	391 - 395
22	26 - 30	126 - 130	261 - 265	396 - 400
23	1 - 5	101 - 105	236 - 240	371 - 375
24	6 - 10	106 - 110	241 - 245	376 - 380
25	11 - 15	111 - 115	246 - 250	381 - 385
Extras	None	226 - 235	361 - 370	496 - 505

TABLE 3
FIVE YEAR STORAGE SCHEDULE

Group	Placed in Storage	Mos. of Storage	Storage Temp	Separator Types
1	Dec 71	60	70	No FSC
2	Dec 71	48	70	No FSC
3	Dec 71	36	70	No FSC
4	Dec 71	24	70	All
5	Jan 72	18	70	All
6	Jan 72	12	70	All
7	Jan 72	9	70	All
8	Feb 72	24	115	No FSC
9	Feb 72	18	115	No FSC
10	Feb 72	12	115	All
11	Feb 72	9	115	All
12	Mar 72	12	160*	All
13	Mar 72	9	160*	All
14	Mar 72	6	70	All
15	Mar 72	6	115	All
16	Mar 72	6	160*	All
17	May 72	3	70	All
18	May 72	3	115	All
19	May 72	3	150	All
20	May 72	2	70	All
21	Jun 72	2	115	All
22	Jun 72	2	150	All
23	Jun 72	1	70	All
24	Jun 72	1	115	All
25	Jun 72	1	150	All

* Reduced to 150° on 19 April 1972

SECTION III

TEST RESULTS

No group of cells survived more than 28 months of the planned five year storage test. Table 4 shows when cell groups were actually removed from storage. Group 9 (115°F) was removed after 15 months storage instead of the 18 months originally scheduled, and Group 8 (115°F) was moved up from 24 to 18 months. Likewise, Group 16 (150°F) was removed at 4 months instead of 6, and Groups 1, 2 and 3 (70°F) were removed at 28 months instead of at 60, 48, and 36 months as originally scheduled. These changes were brought about due to rapid degradation in cell performance prior to the originally scheduled removal times.

In determining capacity loss, the initial capacity was taken to be that capacity obtained from the last conditioning cycle before recharge and storage. The final capacity was that obtained from a single discharge after a period of wet storage. In all instances, the calculated capacity is an average of the capacities of the individual cells of a particular group and separator type.

Table 5 is a summary of the storage data obtained during this program. Where more than one capacity loss figure is listed for any given separator/ temperature/storage time combination, the additional numbers indicate the capacity obtained following recharge and an immediate discharge. For example, cells with three (3) layers of RAI P-2291 exhibited 10% capacity loss after nine (9) months at 70°F. After one recharge in accordance with the charge conditions specified earlier in

TABLE 4

CELL GROUP NUMBERS, STORAGE TIME, AND STORAGE TEMPERATURE

Temp.(°F) \ Storage Time (Months)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	18	20	22	24	26	28
70	23	20	17			14			7			6						5		4		3,2,1
115	24	21	18			15			11			10			9			8				
150	25	22	19	16					13			12*										

* Removed from storage and disassembled. No Capacity Discharge.

TABLE 5

% CAPACITY LOSS AS A FUNCTION OF SEPARATOR, TEMPERATURE, AND STORAGE TIME

Temp (°F.)	SEP*	Storage Time (Months) 1	2	3	4	6	9	12	15	18	24	28
70	4F	4	4	9		10	15/-1	24/-4		28/0	47/4	100
70	2R	1	-2	7		11	59/-5	57/13		100/49	99/83	100
70	3R	5	1	6		2	10/0	26/2		32/-10	53/15	100
70	4R	3	0	1		7	16/-2	16/0		34/0	98/52	100
115	4F	9	19	24		34/-2	47	87/50				
115	2R	9	17	66		100/41	100	100/76	100/70	100/82		
115	3R	2	10	9		37/11	100	100/62	100/38	88/81/24		
115	4R	7	16	18		84/33	100	72/23	100/65	93/88/72		
150	4F	13/-1	61	100/89	100/85		100					
150	2R	22/2	19	54/26	87/45		100					
150	3R	29/-2	45	46/17	100/61		100					
150	4R	19/44	36/31	50/31	56/57		100					

* 4F = 4 Layers FSC
2R = 2 Layers RAE P-2291 (40/60)
3R = 3 Layers RAI P-2291 (40/60)
4R = 4 Layers RAI P-2291 (40/60)

this report, 100% of the original capacity was obtained. Negative numbers indicate a capacity gain.

The data in Table 5 is presented graphically in Figures 1, 2 and 3. It should be stressed at this point that the capacity retention data for any given temperature/separator combination was not obtained from a single group of cells. Each data point corresponds to a particular cell group which was stored for a specified length of time and then discharged to measure capacity. Nothing was done to the cells during their storage periods.

Figure 1 compares the capacity retention of the four separator configurations at 150°F. At this temperature, all separator types show 50% capacity loss in two (2) to four (4) months and 100% capacity loss by the ninth month of storage.

Figure 2 compares the four separator types at 115°F. With respect to capacity retention, two (2) layers of RAI gives the poorest cell performance and four (4) layers of FSC the best. Cells with four (4) layers of RAI appear to give an unusually high capacity after twelve (12) months at 115°F. This capacity, however, is an average of five (5) cells out of which three (3) exhibited no capacity at all.

A comparison of the four separator configurations at 70°F is shown in Figure 3. At this temperature, all separator types behave similarly except for two (2) layers of RAI. This separator begins to exhibit substantial capacity loss after only nine (9) months at 70°F.

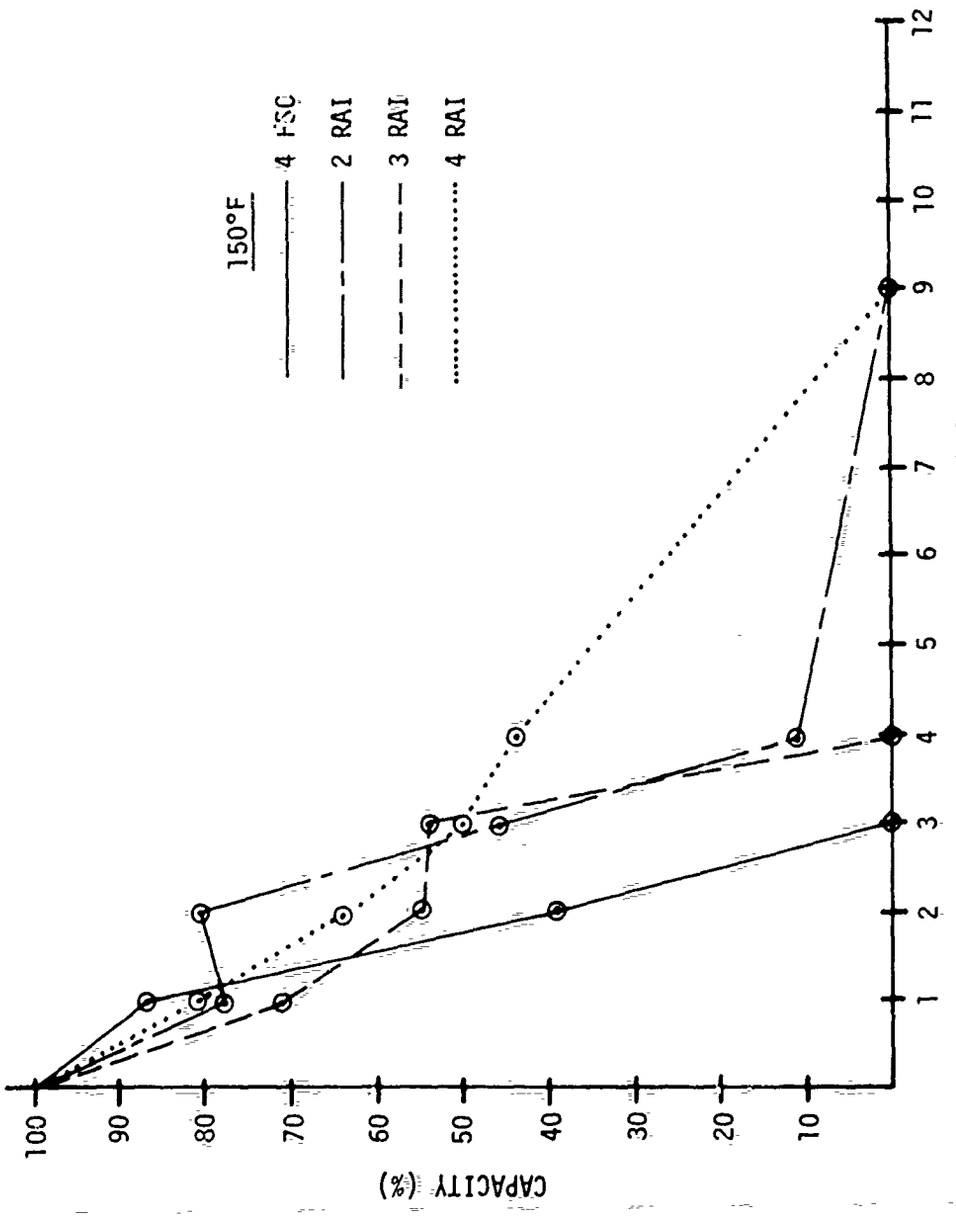


Figure 1. Capacity Retention at 150°F

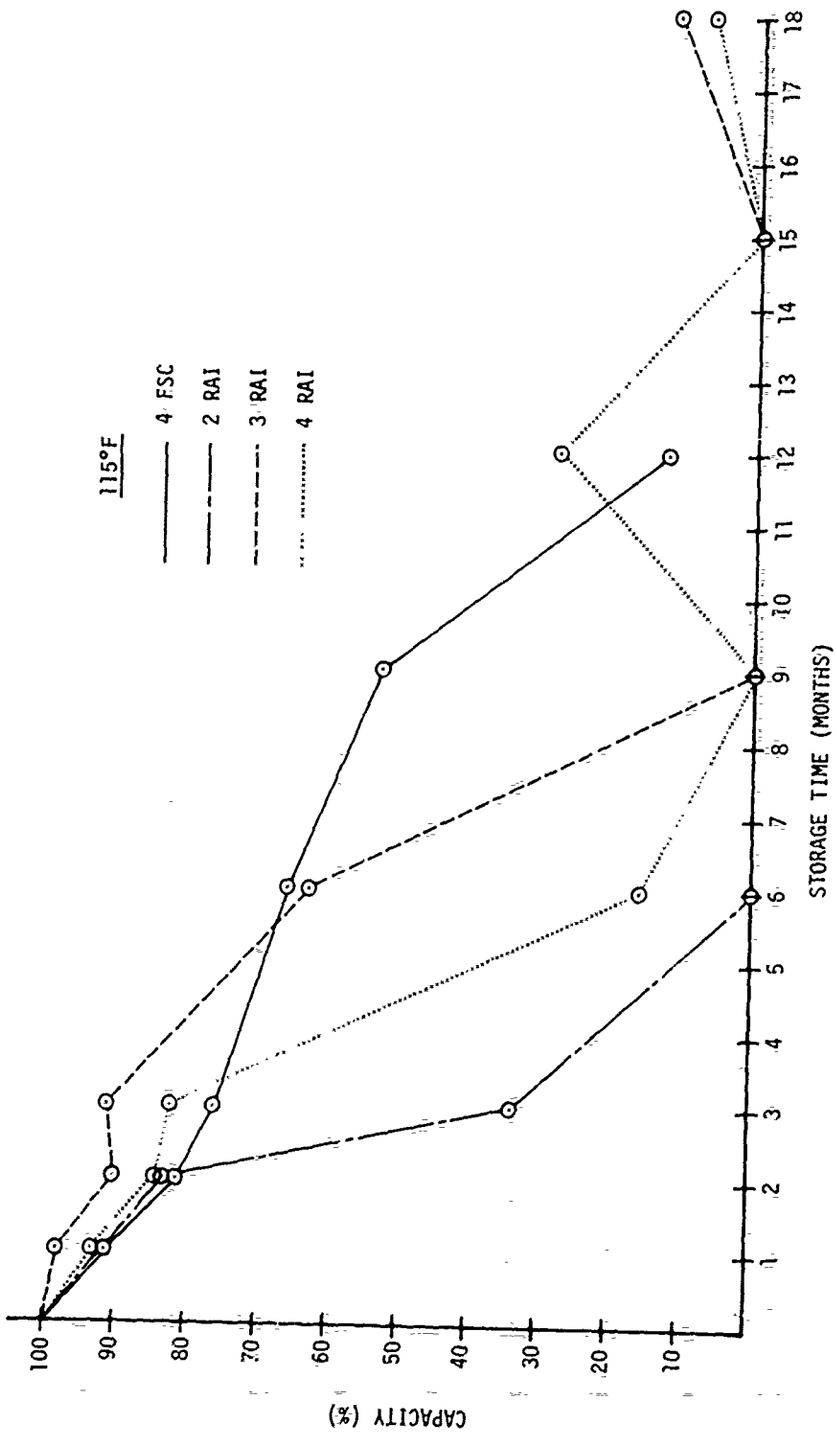


Figure 2. Capacity Retention at 115°F

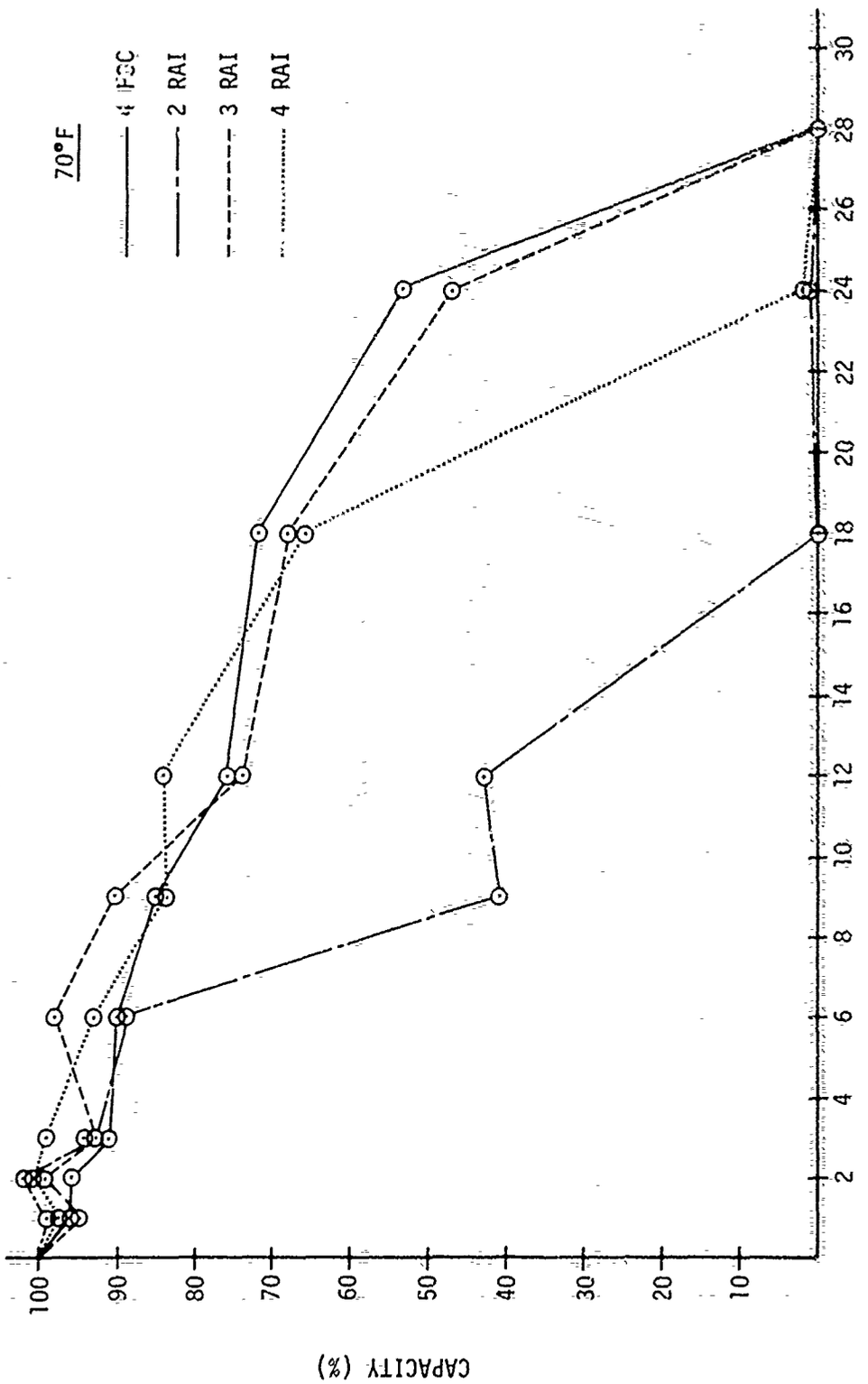


Figure 3. Capacity Retention at 70°F

A possible cause for the fluctuations in capacity retention for the RAI material as opposed to the more linear data for the FSC may be lack of uniformity and difficulty in handling of the RAI material available at the time these cells were built. A manufacturing methods program has since been completed resulting in material which is more uniform and of lower resistance. It has also been discovered that keeping the moisture content to a minimum makes the RAI P-2291 material easier to work with.

As can be seen from Table 6, not all of the capacity loss shown in Figures 1, 2 or 3 was permanent.

At 150°F, 100% of original capacity could be obtained after one (1) month storage with one (1) recharge. The recoverable capacity decreased rapidly to 55% at best after four (4) months.

At 115°F, no recharge data was taken until the sixth month storage point. At this temperature, percent of original capacity obtainable after recharge decreased from a maximum of 100% after six (6) months storage to a maximum of approximately 20% after eighteen (18) months.

At 70°F storage, 100% of the original capacity was recoverable with one (1) recharge after eighteen (18) months storage for all separator configurations except two (2) layers of RAI. Even after two (2) years storage at 70°F, recoverable capacities for three (3) and four (4) layers of RAI were 85% and 96%, respectively.

From the data presented thus far, three (3) and four (4) layers of RAI P-2291 (40/60) appear similar in performance to four (4) layers of FSC. However, an increase in volumetric energy density can be realized by using the thinner RAI P-2291 material. Because only the thickness and not the width or height of the cell stack (plate + separator) is affected by the separator type, the ratio of energy densities of any two (2) separator configurations is equal to the inverse ratio of their cell stack thicknesses. In turn, the cell stack thickness is the difference between the internal cell case depth (dimension D, Appendix I) and the shim thickness used. For cells with four (4) layers of FSC, no shims were used and the cell stack thickness is the actual internal case dimension (1.205"). The 0.250" shim used in cells with three (3) layers of RAI results in a 21% increase in volumetric energy density, and the 0.225" shim used with four (4) layers of RAI yields a 26.5% increase in energy density over cells made with four (4) layers of FSC.

SECTION IV

CELL FAILURE ANALYSIS

Teardown examinations performed on representative samples of all groups disclosed silver accumulation in the separator layers progressing from the positive to the negative plate. As temperature increases, the rates of the processes causing this silver accumulation in the separator increase, resulting in decreased time to shorting and increased rate of capacity loss. In all cases, the cause of ultimate cell failure was internal cell shorting caused by metallic silver accumulation in the separator.

In an attempt to determine the cause of the recoverable capacity loss, an oxygen analysis was performed on the positive plates of several cells according to the procedure shown in Table 6. The four cells chosen for analysis had been stored at 70 F for two (2) years. Two (2) of the cells had four layers of FSC as separator (S/N's 98 and 99) and two (2) had three layers of RAI (S/N's 336 and 339). The results of this analysis are shown in Table 7. Positive plates from cells 98 and 339 were subjected to an oxygen analysis without being capacity discharged. Cells 99 and 336 received a normal capacity discharge followed by a 32 A-H recharge prior to the oxygen analysis.

The positive plate capacities were initially 32 A-H and were assumed not to degrade with storage. Therefore, the capacity remaining in the positive plates after the capacity discharge was determined by subtracting the capacity discharge A-H value from 32. The positive plate capacity

TABLE 6

POSITIVE PLATE OXYGEN ANALYSIS PROCEDURE

- WASH PLATES IN DI WATER TO REMOVE KOH ($\approx$ 2 days)
- DRY IN VACUUM OVEN AT APPROXIMATELY 95°F AND 29 IN Hg
- WEIGH
- SINTER AT 1000°F FOR 30 MINUTES
- WEIGH

TABLE 7
POSITIVE PLATE OXYGEN ANALYSIS RESULTS

- Theoretical Cap (Pos Plate) = 43.9 A-H
- Initial Cap (Pos Plate) = 32 A-H

	4 FSC	3 RAI
• O_2 Analysis of Pos Plate After Stand (w/o Dischg)	(Cell #98) 27.8 A-H	(Cell #339) 34 A-H
	(Cell #99)	(Cell #336)
• Capacity Discharge	19.4 A-H	21.5 A-H
• Capacity Remaining	12.6 A-H	10.5 A-H
• Expected Capacity After 32 A-H Recharge	43.9 A-H	42.5 A-H
• O_2 Analysis of Pos Plate	39.0 A-H	42.5 A-H

of these cells following the subsequent 32 A-H recharge was then the remaining capacity value determined above plus 32.

The oxygen analysis for cells 336 and 339 indicates that, for cells made with three layers of RAI, the positive plate capacity after two years storage at 70°F was still 32 A-H and no appreciable silver had been lost to the separator. The low capacity discharge (21.5 A-H) obtained from cell 336, therefore, must be due to a decrease in the capacity of the negative plate. This capacity is recoverable upon recharge.

The oxygen analysis for cells 98 and 99 shows that, for cells made with four layers of FSC, the positive plate capacity was less than the initial 32 A-H after two years at 70°F but still greater than the capacity discharge value. This indicates that the low capacity discharge obtained from cell 99 (19.4 A-H) was also due to lack of negative plate capacity. The fact that the expected positive plate capacity of cell 99 (43.9 A-H) did not agree with the value obtained from the oxygen analysis (39.0 A-H) indicates that approximately 10 grams of silver had been lost to the separator. (Each cell originally contained 88.5 grams of silver in the positive plate.) Any capacity loss due to silver deposited in the separator would be permanent. As before, however, any capacity loss due to negative plate self-discharge would be recoverable upon recharge.

SECTION V

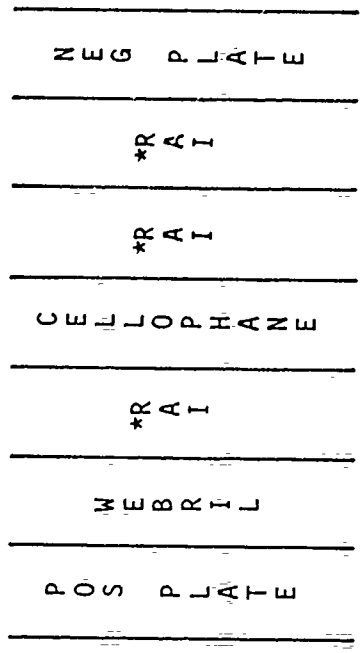
CONCLUSIONS AND RECOMMENDATIONS

Data from this program shows that we do not yet have a separator material which can resist short-circuiting failure due to silver migration at elevated temperatures (115°F and above) in suitable thicknesses to provide high energy densities. However, at 70°F, three and four layers of RAI P-2291 (40/60) are comparable to four layers of FSC with respect to capacity retention with storage. Due to the difference in material thickness, however, a 23% to 26.5% increase in volumetric energy density is realized by using the RAI material in place of the FSC.

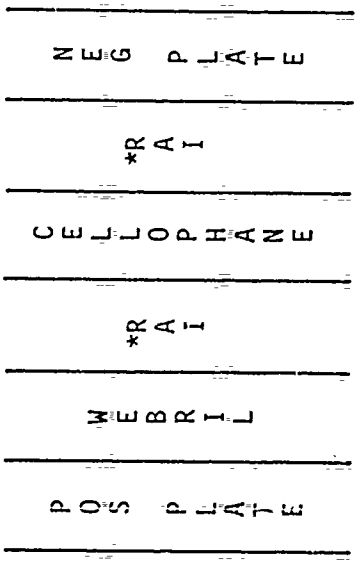
In all cases, the cause of ultimate cell failure was internal cell shorting caused by metallic silver accumulation in the separator. Also, for unshorted cells at room temperature, the recoverable capacity loss is due to negative plate self-discharge. Any non-recoverable capacity loss is due to silver deposited in the separator.

In view of the good capacity retention data obtained at 70°F, it would be desirable to learn how to extend the time to ultimate failure (shorting) as far as possible with the latest material available. It is felt that this could best be accomplished by using laminates of RAI P-2291 and cellophane where the cellophane will act as a sacrificial layer to trap metallic silver and thus delay ultimate failure. This, then, is the objective of Phase II of the Ag-Zn wet stand test. A total of 96 cells are being used to evaluate the two separator configurations

shown in Figure 4. At least one layer of P-2291 is next to the negative plate to resist zinc dendrite penetration, and one layer of P-2291 is between the cellophane and the positive plate to protect the cellophane from rapid oxidation. All of the cells for Phase II were placed in storage by June 1975 with the first cells scheduled to be removed in March of 1976.



CONFIGURATION B



CONFIGURATION A

*RAI P-2291 (40/30)

Figure 4. Phase II Separator Configurations

APPENDIX

CELL COMPONENT DESCRIPTION

POSITIVE PLATE: (8 per cell)

Dimensions - 1.844" X 2.50" X 0.034"

Loading - 2.40 gm Ag/sq. in.

Theoretical Cap - 43.9 A-H

NEGATIVE PLATE: (9 per cell)

Dimensions - 1.844" X 2.50" X 0.065"

Loading - 2.70 gm ZnO blend/sq. in.

Theoretical Cap - 73.8 A-H

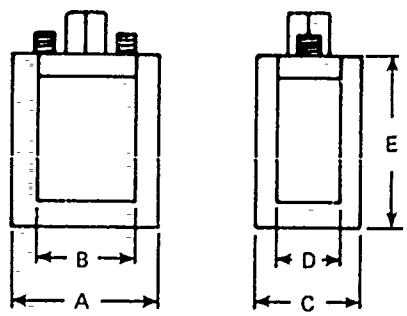
SEPARATOR THICKNESS:

Webril (E-1408)	- 0.001"
FSC (dry)	- 0.0035"
FSC (wet)	- 0.007"
RAI P-2291	- 0.001"

SHIM THICKNESS (TOTAL):

4 Layers FSC	- No Shim
4 Layers RAI	- 0.225"
3 Layers RAI	- 0.250"
2 Layers RAI	- 0.275"

CASE DIMENSIONS:



A = 2.313" B = 2.192"

C = 1.320" D = 1.205"

E = 3.79"

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